
WORKSHOP ON
Advanced Electron
Microscopy in
Materials Physics

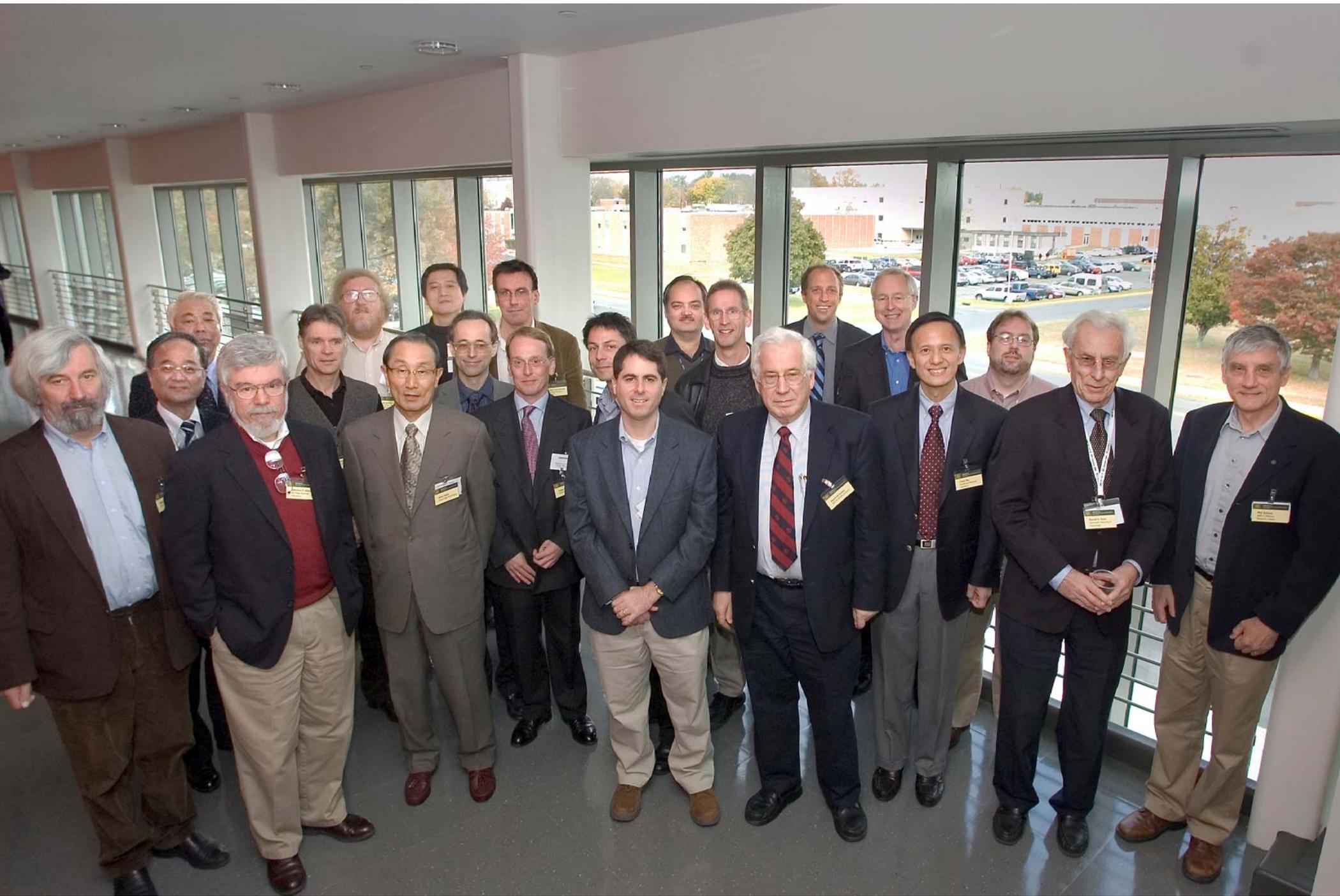
Nov. 7-8, 2007

Long Island, New York



WORKSHOP ON
Advanced Electron Microscopy in Materials Physics
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Sponsored by the Institute for Advanced Electron Microscopy,
Center for Functional Nanomaterials, and Hitachi High Technologies.



ADVANCED ELECTRON MICROSCOPY AND MATERIALS PHYSICS WORKSHOP

November 7-8, 2007
Building 735, CFN Seminar Room
Brookhaven National Laboratory
Long Island, New York

PROGRAM

WEDNESDAY (November 7th)

7:30 – *Continental Breakfast*

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8:30 - Welcome from Organizers – Yimei Zhu (BNL) & Konrad Jarausch (Hitachi)

8:40 - Opening Remarks – Emilio Mendez, CFN Director

Instrumentation & Aberration Correction

Rudolf Tromp, Session Chair

8:45 - Harald Rose (TU-Darmstadt) – The History of Aberration Correction

9:15 - Max Haider (CEOS) – New Correctors to Fulfill the Requirements of Future Instruments

9:45 - Bernd Kabius (ANL) - Aberration Correction Concepts Within the TEAM Project

10:15 *Coffee Break*

10:30 - Phil Batson (IBM) - Unexpected Benefits from Aberration-Corrected Electron Optics

11:00 - Rodney Herring (Victoria) - Coherence Property Measurements of Phonon-, Plasmon-,
and Ionization-loss Electrons and Their Contributions to the Stobbs Factor

11:30 - Manfred Ruehle (MPI-Stuttgart) – Challenges of Electron Microscopy in Materials
Physics

12:00 - *Lunch*

Advanced EM in Materials Physics

Manfred Ruehle, Session Chair

1:30 - Steve Pennycook (ORNL) - New Views of Materials with Aberration-Corrected STEM

2:00 - Koji Kimoto (NIMS) - A Few Practical Aspects of Atomic-Column Imaging Using ADF
and EELS

2:30 - Nigel Browning (UC-Davis) - Imaging and Spectroscopy of Nanoscale Systems in the
STEM

3:00 - *Coffee Break*

3:30 - Yimei Zhu (BNL) – Structural Analysis of Layered Thermoelectric Oxides Using
Aberration Corrected STEM/EELS

- 4:00 - Ji-Jung Kai (National Tsing Hua University) - The Characterization of Helium Bubbles in SiC/SiC Composite Using EELS and the Current Development on Structure Reversion
- 4:30 - Rudolf Tromp (IBM) - Cathode Lens Microscopy: The Next Generation
- 5:00 - Ray Twesten (Gatan) - Advances in Multi-Dimensional Imaging Techniques for STEM
- 6:00 - *Reception, Berkner Hall Lobby*
Greetings from Hitachi – I. Muta (General Manager, Sales) and Y. Kawasaki (Executive Vice President)
- 7:00 - *Dinner, Berkner Hall Cafeteria*
Talk by Konrad Jarausch (Hitachi) – Novel Approaches for In-Situ and 3D Microscopy

THURSDAY (November 8th)

7:30 – *Continental Breakfast*

Advanced EM in Materials Physics

Mike O’Keefe, Session Chair

- 8:00 - Robert Klie (UI-Chicago) - Atomic-Scale Studies of Complex Oxide Interfaces Using Aberration-Corrected Z-contrast Imaging and EELS
- 8:30 - Angus Kirkland (Oxford) - Imaging and Exit Wave Reconstruction in an Aberration-Free Environment
- 9:00 - Rafal Dunin-Borkowski (TU Denmark) - Catalyst Nanoparticles Imaged Using Aberration Correction, Focal Series Restoration and Electron Tomography
- 9:30 - David Muller (Cornell) - Atomic-Scale Chemical Imaging by Aberration-Corrected Microscopy
- 10:00 – *coffee break*
- 10:15 - Mike O'Keefe (LBNL) - Cs Correction and Focal-Series Reconstruction of the Exit-Surface Wave
- 10:45 - Jim Bentley (ORNL) - Nanoscale Elemental Mapping in TEM and STEM
- 11:15 - Larry Allard (ORNL) - Catalyst Characterization via Aberration-Corrected STEM in the HTML User Program
- 11:45 - Doug Blom (U-SC) - Aberration-Corrected STEM of MoVTaNbO Complex Oxide Catalysts.
- 12:15 - Closing remarks – Yimei Zhu (BNL) + Konrad Jarausch (Hitachi)

12:30 - *Lunch*

CFN instrumentation, small group discussion and tours (options):

- 1:30 - Operation and optimization of Cs corrected STEM/EELS (in laboratory) and tour of CFN facilities and instrumentation – break into small groups
- (a) Live Cs-corrected STEM/EELS at CFN and discussion (60 minutes)
- (b) Tour of the CFN facilities + microscopes and discussion (60 minutes)

ABSTRACTS

DAY ONE

History of Aberration Correction

Harald Rose

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Aberration correction in electron microscopy dates back to Otto Scherzer who proved in 1936 that chromatic and spherical aberrations of rotationally symmetric electron lenses are unavoidable [1]. His finding was so important that it was named “Scherzer theorem”. In 1947, Scherzer found an ingenious way for enabling aberration correction. He demonstrated in a famous article that it is possible to eliminate chromatic and spherical aberrations by lifting any one of the constraints of his theorem, either by abandoning rotational symmetry or by introducing time-varying fields, or space charges [2]. Moreover, he proposed a multipole corrector compensating for the spherical aberration of the objective lens. Subsequently, his student R. Seeliger built and tested this corrector. However, it was G. Moellenstedt who first demonstrated experimentally the correction of spherical aberration by means of this corrector [3]

In 1964, Deltrap built a telescopic quadrupole-octopole corrector to eliminate the spherical aberration of a probe-forming lens. Although he nullified this aberration, he failed, like his predecessors, to improve the actual resolution of the uncorrected system because at that time the resolution was limited by mechanical and electrical instabilities rather than by the defects of the electron lenses. In 1971, H. Rose proved that all known correctors introduce large coma and proposed a novel aplanatic corrector utilizing symmetry properties. This corrector was built and tested successfully in a test microscope within the frame of the so-called Darmstadt project and it demonstrated for the first time the simultaneous correction of chromatic and spherical aberration. The project was abandoned after the death of O. Scherzer, although it was successful as far as it went.

Experimental work on aberration correction was resumed by M. Haider in 1992 within the context of the “Volkswagen Project” aimed to improve the resolution limit of a 200 kV transmission electron microscope (TEM) by eliminating spherical aberration and coma by means of a highly symmetric aplanatic hexapole corrector [4]. Together with S. Uhlemann he developed a fast alignment procedure, which enables a precise and fast elimination of the spherical aberration and all resolution-limiting parasitic aberrations originating from mechanical inaccuracies, magnetic inhomogeneities and misalignment. Employing this computer-assisted alignment, M. Haider improved in 1997 for the first time the performance of a TEM by reducing the resolution limit from 2.1Å to 1.4Å [5]. In the meantime this limit has been lowered further to about 0.8Å

The successful correction of chromatic and spherical aberration of a low-energy electron microscope by means of an electron mirror has been demonstrated recently by T. Schmidt for the SMART microscope by reducing its resolution limit from 15nm to 3nm.

References:

¹O. Scherzer, Z. Physik **101** (1936) 593.

²O. Scherzer, Optik **2** (1947) 114.

³G. Moellenstedt, Optik **13** (1956) 209.

⁴H. Rose, Optik **85** (1990) 19.

⁵M. Haider, H. Rose, S. Uhlemann, E. Schwan, H. Kabius, K. Urban, Nature **392** (1998) 768.

New Correctors to Fulfill the Requirements for Future Instruments

M. Haider, H. Mueller, S. Uhlemann, J. Zach and P. Hartel
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The improvement of the resolving power of a TEM and a STEM by means of the correction of the unavoidable spherical aberration was a long ongoing effort. Several attempts to correct these main aberrations have already been made since the first proposal of O. Scherzer in 1947 [1] on how to correct the axial aberrations of an objective lens. However, just with the emergence of the first successfully Cs-corrected TEM [2] the gate for the development of a new generation of high resolution TEMs and STEMs has been opened. Meanwhile Cs-correctors exist for the main class of high resolution TEM and STEM for an accelerating voltage range from 80 kV up to 300 kV. The EM manufacturers are offering these Cs-corrected TEMs and/or STEMs for high resolution and analytical purposes. The resolution of these Cs-corrected lenses is now limited by incoherent aberrations which cannot be compensated by a Cs-correction system. The main incoherent aberration is the chromatic aberration for which the product of $E \cdot C_c$ is the important parameter. This product defines the information limit and, hence, the attainable resolving power of an instrument. Besides this chromatic aberration one also has to consider other disturbing factors such as instabilities of electronics or external sources of noise.

For further improvements of the attainable resolving power one has to investigate the limiting parameters. We are only concentrating on the instrumental parameters which are:

- The wave length (or energy E) of the electrons,
- The energy width ΔE of the electrons one would like to image or to focus into a small probe.
- The chromatic aberration coefficient C_c of the objective lens one uses.
- The higher order aberrations of the objective lens,
- Any incoherent disturbance introduced by the instrument or by the environment.

Current and future developments of aberration correctors are dependent on the system one aims for:

- For UHR-STEM one has to reduce the higher order aberrations, especially the fifth-order aberrations in order to achieve an illumination cone of more than 40 mrad. This has just been currently done for a high resolution STEM [3] for which the goal is to achieve in combination with a monochromator a probe size of $d = 0.05 \text{ nm}$ ($d \sim 20 \text{ \AA}$).
- A C_c -correction system for a high resolution TEM for which a resolving power of 0.05 nm and a large field of view should be achieved. This project is in good progress and the first results should be obtained in the first half of 2008.
- The chromatic aberration of a high resolution probe forming system can be compensated by an appropriately designed C_c/C_s -corrector.
- The off-axial coma of a high resolution TEM can also be corrected for and this would lead to a Cs-corrected TEM with a large field of view.

References

¹O. Scherzer, *Optik*, 2, (1947) 114.

²M. Haider et al., *Nature*, 392, (1998) 768.

³H. Müller, S. Uhlemann, P. Hartel and M. Haider, *Micr. & Microanal.* 12 (2006) 442-455

Aberration Correction Concepts Within the TEAM Project

B. Kabius

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Argonne, IL 60439

During the last 10 years several aberration-correction concepts for electron microscopes have succeeded in improving spatial resolution and analytical capabilities. Electron optical systems for correction of spherical aberration are now a valuable tool for material science research and several investigations have already exploited some of the benefits of C_s -correction for high-resolution TEM and STEM. The TEAM project is a collaborative DOE project which will extend the present capabilities of aberration correction technology. The goals for aberration correction within the TEAM project are:

- Correction of higher order aberrations such as fifth order spherical aberration is required for improving interpretability at sub-Angstrom resolution (TEM) and higher beam currents in smaller electron probes (STEM).
- Improving the information limit to 0.5\AA by correction of chromatic aberration (C_c) and energy monochromation.

The information limit can be improved alternatively by reducing the energy width of the electron emitter using a monochromator. One of the disadvantages of this concept is a strong loss of brightness due to the monochromator. Correction of chromatic aberration improves the damping envelope of temporal coherence without the need for a monochromator thus achieving higher contrast transfer and higher beam currents.

Lens systems for the correction of chromatic aberration for TEM or STEM have not yet been implemented because of the stringent current stability requirements of about 10^{-8} for the multipole elements. Recently, new designs for chromatic aberration correction have been suggested by H. Rose and were analyzed and refined within the TEAM project. At present a corrector is under development which is according to simulations capable of correcting C_c for acceleration voltages up to 300kV. Furthermore spherical aberration as well as axial aberrations and off-axial coma will be corrected up to the fifth order thus enabling aberration free imaging up to a resolution of 0.5\AA for a large field of view of $2000*2000$ independent image points in TEM mode which is one of the goals of the TEAM project.

Based on this design study and recent progress improving the stability of power supplies C_c correction appears to be feasible with current technology. Further applications of C_c -correction for EFTEM and *in situ* experiments will be discussed.

This work was supported by the US Department of Energy, BES-Materials Sciences, under Contract W-13-109-ENG-38.

Unexpected Benefits from Aberration-Corrected Electron Optics

P.E. Batson

IBM Thomas J. Watson Research Center, Yorktown Heights, New York 10598

Without a doubt, aberration correction electron optics has vastly changed the quality and accuracy of obtainable imaging data, particularly in the lower voltage microscopes. The ability to image very small crystallites, and to resolve defect structures was expected and welcomed. But with any new tool, we also find that there may also be unexpected benefits. For instance, with a sub-Å probe size, single atom contrast is so high that images can be taken very rapidly, allowing time-dependent inspection of very small objects. Since the electron beam interaction with atoms can give up a fraction of an eV in energy, there is a good chance that single atoms will move under the beam, allowing measurement of an ensemble of atomic positions available to that atom. Thus, some types of dynamic experiments can be done if the probe size, current and dwell time is controlled. In the future, precise control of aberrations implies an ability to control the position and phase of incident and scattered *wavefunctions*. This, in turn implies that we will have much more detailed information about the specimen excitation created by an inelastic scattering event than has been accessible in the past. This should allow, for instance, measurement of structural anisotropy using EXELFS on an atom column by column basis. It also seems possible that very accurate control of the position and propagation direction of the incident and final beam *wavepackets* may allow us to use non-zero impact parameter scattering to probe specific anisotropic specimen properties.

Coherence Property Measurements of Phonon-, Plasmon-, and Ionization-loss Electrons and Their Contributions to the Stobbs Factor

Rodney Herring

University of Victoria Canada

Energy-filtered electron holography of diffracted beams has been used to measure the degree of coherence of energy-loss electrons. Since the fast electrons of the primary beam when passing through the material create phonons, plasmons, magnons, etc., the energy-loss electrons retain the coherence properties of these quasi-particles such as their lateral coherence width, which is required for understanding nanoscience and for our development of nanotechnology. Interestingly, the ability to measure the intensity of ionization-loss electrons will enable the creation of a new magnetic microscopy method, as well as, the coherence properties of the magnon. This work has also led to a better understanding of the Stobbs factor, which is a contrast mismatch between experimental lattice images and simulated lattice images that is required for the development of high-resolution quantitative lattice imaging where the type and number of atoms making up a lattice image can be determined. A mathematical expression is derived that adequately predicts the Stobbs factor.

Challenges of Electron Microscopy in Materials Science

Manfred Ruehle, Christoph Koch, Wilfrid Sigle, Peter van Aken
Max Planck Institute for Metals Research, Stuttgart/Germany

The properties of materials depend on their microstructures. An important task of materials science (materials physics) is to reveal the correlation between microstructure and therelevant property. This, in turn, requires the analysis of defects in the materials or materials systems. Defects can be distinguished according to their dimensionality: 0-dimensional (point defects), 1-dimensional (dislocations), and 2-dimensional (interfaces) defects. It is crucial that the defects are characterized and their critical parameter be determined.

Different TEM techniques allow the determination of the structure, and composition of the defect and its surrounding. Qualitative and semi quantitative results can be obtained rather easy. A quantitative evaluation (Q-TEM) requires the determination of the desired value including error bars (reliabilities). Q-TEM is often very time consuming and is, unfortunately, not pursued in many laboratories. It is however essential that Q-TEM is being done to solve critical problems.

In the talk several examples will be presented, mainly for grain boundaries (in metals and ceramics) and interfaces (between metals and oxides). Most results obtained so far (in our laboratory) do not possess the necessary accuracy so that they can be used to a critical comparison with results obtained by modelling using different approaches.

Advanced instrumentation (aberration corrected instruments with high stabilities) and better TEM specimens will lead to experimental results with better reliability.

New Views of Materials Through Aberration-Corrected STEM

S. J. Pennycook^{1,2}, M. Varela¹, M. F. Chisholm¹, A. Y. Borisevich¹, A. R. Lupini¹, K. van Benthem^{3,1}, M. P. Oxley¹, W. Luo^{1,2}, S-H. Oh^{1,4}, D. Kumar^{4,1}, D. L. Sales⁵, S. I. Molina⁵, A. G. Marinopoulos², and S. T. Pantelides^{2,1}

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The aberration-corrected STEM provides Z-contrast images and electron energy loss spectroscopy with resolution near or below 1 Ångstrom and 0.5 eV, respectively, which represents unprecedented sensitivity for determining atomic arrangements, impurity sites and local electronic structure in materials. For example, charge carriers can be located within the unit cell of the high-temperature superconductor YBCO, and charge transfer across a superconductor/ferromagnet interface can be quantified and linked to the material's properties. Similarly, column-by-column compositional mapping of InAsP quantum wires coupled to elasticity calculations explains their growth morphology and optical properties. The aberration-corrected STEM also has a much decreased depth of field, which can be used to provide structural information in three dimensions by optical sectioning, similar in principle to confocal optical microscopy. Individual Hf atoms have been located within a Si/SiO₂/HfO₂ gate dielectric structure to a precision of 0.1 x 0.1 x 1 nm, and the perturbed electronic structure linked by density functional theory to macroscopic device properties. Individual gold atoms have been imaged inside Si nanowires in substitutional and interstitial configurations, with number densities that are in order of calculated formation energies.

This research was sponsored by the Office of Basic Energy Sciences, Division of Materials Sciences and Engineering and by appointment (MPO, KvB) to the ORNL Postdoctoral Research Program administered jointly by ORNL and ORISE.

A Few Practical Aspects of Atomic-Column Imaging Using ADF and EELS

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Atomic-column resolution in STEM and EELS has been recently achieved by a few authors [1]. We have also demonstrated atomic-column imaging of $(\text{La,Sr})_3\text{Mn}_2\text{O}_7$ and Si_3N_4 [2]. Here, we discuss a few practical aspects of atomic-column imaging; the atomic-site dependence and the energy-loss dependence of the spatial resolution are elucidated on the basis of the experimental results and multislice calculations. We describe two factors for realizing atomic-column imaging in terms of localization in elastic and inelastic scattering. One is the channeling of the incident probe due to dynamical diffraction [3], which has atomic-site dependence. The other is the localization in inelastic scattering [4]; in addition to the energy-loss dependence of delocalization, we point out its dependence on the offset energy from the ionization energy, *i.e.*, an additional localization factor concerning the Bethe surface. The present atomic-column observations indicate that the incoherent EELS imaging, which can be interpreted intuitively, is achievable under appropriate experimental conditions, such as high energy-loss, a small convergence angle and a large collection angle.

¹M. Varela, *et al.* PRL 92(2004) 095502; M. Bosman, *et al.* PRL 99(2007) 086102; M.P. Oxley, *et al.* PRB 76(2007) 064303.

²K. Kimoto, *et al.* Micron (in press); K. Kimoto, *et al.* (accepted).

³J. Fertig and H. Rose Optik 59(1981) 407; R.F. Loane, *et al.* Acta Cryst. A 44 (1988) 912.

⁴S. J. Pennycook Contemp. Phys. 23(1982) 371; D. A. Muller and J. Silcox Ultramicrosc. 59(1995) 195; R. F. Egerton Micron 34(2003) 127; M.P. Oxley, *et al.* PRL 94(2005) 203906.

Imaging and Spectroscopy of Nanoscale Systems in the STEM

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The recent developments of aberration correction and monochromation have given the scanning transmission electron microscope (STEM) unprecedented new capabilities to investigate the atomic and electronic structure of nanoscale materials. In particular, aberration correctors have pushed spatial resolution into the deep sub-angstrom regime while monochromators are pushing spectral resolution rapidly towards the sub-100meV level. However, these capabilities also present challenges for the interpretation and routine quantification of experimental images and spectra. In the case of the aberration correctors, increases in beam current mean that utilizing the same experimental procedures as have been traditionally used for materials science quickly leads to electron beam modification of the sample. In the case of the monochromators, the resolution of the spectrum means that care must now be taken in interpreting fine-structure, especially in the low-loss region of the spectrum. Here, examples of the types of experiments that can readily be performed with aberration correctors and monochromators will be presented. Emphasis will be placed on recent results from UC-Davis, LLNL and LBNL showing the application of these methods to understanding the properties of quantum dots and quantum wells. The potential to extract information from “low-dose” STEM experiments will also be highlighted by the analysis of a series of nanoscale catalysts particularly susceptible to electron beam modification and particle motion.

Structural Analysis of Layered Thermoelectric Oxides Using Aberration Corrected STEM/EELS

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⁴ Gatan, Inc. Pleasanton, CA 94588

Cobaltates are of great interest due to its unique thermoelectric and magnetoresistance properties and practical energy-related thermal power applications. In order to understand the origin of these physical properties of layered $\text{Ca}_3\text{Co}_4\text{O}_9$, more precisely, $[\text{Ca}_2\text{CoO}_3]_{0.62}\text{CoO}_2$, cobalt oxides that exhibits high thermoelectric power, a clear understanding of the crystal and electronic structure of the materials is required. $[\text{Ca}_2\text{CoO}_3]_{0.62}\text{CoO}_2$ crystal has a complex misfit lattice structure with rock-salt-type $[\text{Ca}_2\text{CoO}_3]$ slabs and interpenetrating CdI_2 -type $[\text{CoO}_2]$ layers stacked along the c -axis with an incommensurate modulation along the b -axis. The structure can be approximated as a 3D commensurate cell containing 8 $[\text{CoO}_2]$ motifs along the b -axis that stack along the c -axis with 5 $[\text{Ca}_2\text{CoO}_3]$ blocks.

Single crystal and powder samples were analyzed using electron diffraction including electron diffuse scattering, HRTEM, and synchrotron x-ray. A 4-dimensional (4D) structure model was proposed first and then refined by Rietveld algorithm using a 4D super-space approach in order to take the incommensurate modulation into account. To further observe the possible difference in electronic and thermal conductivities of the CoO_2 and the CoO layers (electron crystal and phonon glass) in the $[\text{CoO}_2]$ and $[\text{Ca}_2\text{CoO}_3]$ motifs, respectively, simultaneous acquisitions of column-by-column scanning transmission electron microscopy (STEM) and electron energy-loss spectroscopy (EELS) were carried out using state-of-the-art aberration corrected microscopes, in particular the new Hitachi HD2700. Emphasis was placed on the local lattice order and disorder, variation in oxygen-hole concentration, cation vacancies, bonding states, and valencies across the misfit layers as well as within the layers. Significant in-plan lattice displacement and modulation were observed in atomically resolved STEM-ADF (annular-dark-field) imaging and the results were compared with ADF image simulation. By analyzing the O K-edge prepeak intensity and intensity ratio of the Co-L_{3,2} edges we observed significant charge transfer between the rock-salt CoO layer and CdI_2 -like $[\text{CoO}_2]$ layer. The EELS data agree well with out DFT based electronic structure calculations.

The authors would like thank J. Qing, and Q. Li for providing samples and J. Hanson for collecting x-ray data at NSLS. The work at BNL was supported by U.S. DOE, BES (DE-AC02-98CH10886).

The Characterization of Helium Bubbles in SiC/SiC Composite Using EELS and the Current Development on Structure Reversion

Ji-Jung Kai and Fu-Rong Chen

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Nuclear reactor core is a very crucial environment for structural materials, not only the high temperature and corrosive atmosphere but also very high flux of fast neutrons which induce severe radiation damage in the materials. For advanced nuclear power reactor, in order to reach higher thermal efficiency, we need to have much higher coolant temperature (1000° C) which is a even great challenge to structural materials. SiC/SiC composites are one of the candidate materials for the fusion reactor first wall and blanket materials because of the very high temperature mechanical strength (above 1000°C) and the low activation nature of them. In order to simulate the D-T fusion reactor environment, triple -ion-beam simultaneous irradiation (Si^+ , He^+ , and H^+) was performed to simulate high energy neutrons, helium ions, and deuterium and tritium ions, respectively. The major microstructural evolution includes the formation of He bubbles and voids which may affect the mechanical property of the materials. For understanding the fundamentals of the role of He and D and T in the formation of these cavities, it is important to measure the density of He, or D and T in the bubbles and in the matrix. This phenomenon was studied in detail by using high resolution transmission electron microscopy (HRTEM) with electron energy loss spectroscopy (EELS). We did careful measurements on the blue shift of the He K-edge with regard to the diameter of He bubbles which in turn relates to the density of He (also pressure) in the bubbles. Using this method, we can precisely determine the amount of He inside the bubbles and in the matrix of SiC. Similar method was used for hydrogen measurement. Too. Nevertheless, due to it's very high mobility at this temperature range, we did not find H-peak in the EELS spectrum either in the bubbles or in the matrix. However, from the distribution of bubble density and diameter, we concluded that hydrogen played an important role in the nucleation stage to stabilize the bubble which in turn enhanced the number density of bubbles.

We have started to work on structure reversion from the exit wave by using both “Reverse Mutislice Method” and “Reverse Channeling Method”. The final goal for this is to achieve atomic resolution tomography. Two cases were studied, namely a simulated NiSi_2 example and an experimental SrTiO_3 case. From the reverse multislice method, the simulated wedge-shaped NiSi_2 can be retrieved completely. From the reverse channelling method, it also shows close results to the actual number of Ni and Si atoms. The results for the experimental SrTiO_3 [110], both methods showed a reasonable agreement. The number of atoms in each of the atomic columns is in a difference of one atomic layer. However, without an exit wave from another view direction, it is too soon to conclude the exact result. It is believed that both the reverse multislice method and the reverse channelling method are having the potential to retrieve the structure information.

There are three bottlenecks in bio-imaging using electron microscope. First of all, the bio-sample is usually composed of light elements that interact very weak with electron and such that give rise to weak contrast in the image. Second, the TEM has to be operated in vacuum, therefore, no live sample is allowed and the bio-sample usually is dried out before it is put into TEM system.

Finally, the bio-sample usually suffers from the radiation damage under the high energy electrons during experiment.

In order to overcome the above obstacles for bio-imaging using TEM and its related analytical techniques, The bio-imaging center supported by NSC, IOP, academic Sinica and NSRRC was funded to develop an TEM that allows us to study phase contrast of live bio-samples. In our proposed TEM system, the live bio-sample can be studied in a sealed wet environment with the combined capability of obtaining phase contrast. The preliminary results of the wet cell and electrostatic phase plate will be shown in the presentation. The partial coherent imaging theory of phase contrast from electrostatic phase plate is being developed. An iterative algorithm and fractional Fourier transformation to recover the pure phase image. This method may reduce the number of images needed for reconstructing the “phase” tomography”.

Cathode Lens Microscopy: The Next Generation

Rudolf M Tromp

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Low Energy Electron Microscopy (LEEM) and Photo Electron Emission Microscopy (PEEM) are cathode lens instruments that have become useful research techniques only during the last twenty years.

LEEM is used mostly to study the structure of surfaces and interfaces, relying on contrast mechanisms derived from Low Energy Electron Diffraction. PEEM on the other hand uses the photoelectron emission process to obtain spatially resolved spectroscopic information on the valence band, core levels, or magnetic structure.

Typically, spatial resolution is limited to 5-10 nm in LEEM, and 10-50 nm in PEEM.

While aberration correction is now becoming well-established in transmission electron microscopes, there is still significant room for development of aberration correction in LEEM and PEEM instruments.

Proof of principle of mirror-based correction of C_s and C_c has been established recently.

Additionally, inclusion of an electron energy filter greatly enhances the analytical capability of the instrument, but at the expense of significantly increased instrument complexity.

In this talk I will present results obtained recently with a much simplified imaging electron energy filter which, provides excellent energy and spatial resolution with the simple addition of a strategically placed slit in a standard LEEM instrument. I will also present the design of a new aberration corrected LEEM/PEEM instrument with a compact footprint, straightforward design, and a spatial resolution in the 2-3 nm range. This instrument is presently under construction and is expected to come online in 2008.

Finally, I will discuss the development and installation of new light sources that make lab-based spectroscopic PEEM a reality.

Advances in Multi-Dimensional Imaging Techniques for the Modern Scanning TEM

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Computer automation, high-efficiency spectrometers, and high-speed cameras in combination with modern electron microscopes allow the routine acquisition of information rich, multi-dimensional data sets in a simplified, automated manner. These multi-dimension data sets run the gamut from straight forward, line scans of EELS spectra to exotic combinations of spectral, real-space and k-space information. Continuing advances in computing power and processing tools enable rapid identification and extraction of the elemental, chemical and physical information contained within these data sets. Such improvements have made these rich data collection and analysis techniques available to nearly all characterization laboratories.

While software and automation allows routine acquisition of these data sets, obtaining quality data requires the understanding of the limits and trade offs inherent in the experiment. We will reference several case studies that serve to illustrate the strength and potential pit falls of these techniques with particular emphasis on the simultaneous acquisition and analysis of complementary techniques such as EELS and EDS spectrum imaging, spatially resolved diffraction, and electron tomography.

Novel Approaches for In-situ and 3D Microscopy

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³ Hitachi High Technologies, Japan

The nanoscale study of complex 3D geometries and in dynamic processes is driving the continuing development of instrumentation and methodology. New tools are required for characterizing nanoparticles with complex three-dimensional (3D) structures and properties. Furthermore the study of nanoparticles in different ambient temperatures and partial pressures is required to understand how structure-property relationships are effected by their environment. Electron microscopy techniques are widely used to characterize morphology, composition and electronic properties of materials at the atomic or near-atomic scale but are not well suited for analyzing complex 3D structures or samples in different ambients. A nanoparticle must be either tilted or rotated with respect to the electron beam to enable 3D analysis but this is often limited by STEM/TEM pole-piece geometry. The high vacuum of a TEM/STEM also precludes the study of living specimens or specimens in a gas or liquid environment. Here we report recent advances in instrumentation and methodology to overcome some of these limitations.

A novel rotation holder has been developed [1] which allows analysis from a full 360 degrees as opposed to traditional tomographic tilt-series based analysis which can suffer from missing wedge artifacts [2]. Focused ion beam (FIB) based techniques can be used for site-specific sample preparation and for attaching samples to such rotation holder stubs. A new protocol for functionalizing these sample holders has been developed to enable 360° TEM/STEM observation of individual nanoparticles and nanostructures [3]. The three step process includes FIB milling to customize sample stub geometry, thin film deposition for substrate selection and subsequent chemical functionalization for nanoparticle adhesion. This protocol was used to determine the morphology and local material properties of individual Au/SiO₂ core-shell nanoparticles used in a DNA detection assay.

A new 300kV TEM has been developed for atomic resolution studies of gas-solid reactions at elevated temperatures. This unique design combines a LaB₆ source with differential pumping aperture only above the sample to provide a unique combination of imaging performance and in-situ capability. The LaB₆ source is much less sensitive to vacuum fluctuations than a field emission source, and allows for interface and boundary imaging with fewer delocalization effects. Gas can be introduced directly into the specimen chamber while samples are heated up to 1500 °C to activate the gas–solid interaction. The stability and gas-injection design of the sample holder allows these interactions be studied at atomic resolution [4]. The microscope is equipped with a high-speed CCD and has been used to capture real-time movies of layer-by-layer growth of nanoparticles. Several examples will be shown of how this low-cost E-TEM is being used to support the development of catalysts and nano-materials.

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Hitachi HD2700C 200kV CFE Cs corrected STEM

Cold Field Emission electron source (Hitachi patent):

0.35eV energy resolution, in a 2A spot with 200pA of beam current!

Cs corrected STEM (integrated CEOS corrector):

1A HAADF resolution, >40mrad illumination semi-angle

EELS performance (new Gatan HR-Enfina design):

ultra-fast spectrometer with 3rd order correction (300 spectra/sec)

Diffraction analysis (Gatan 2.6k Orius Camera)

nanobeam (NBD) + convergent beam diffraction (CBED)

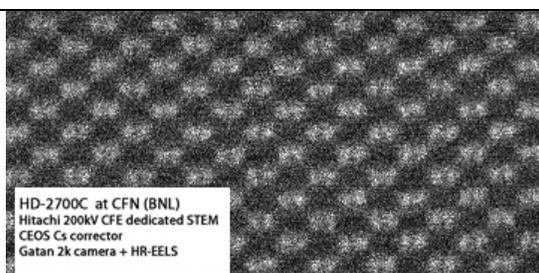


HD-2700C now installed at BNL

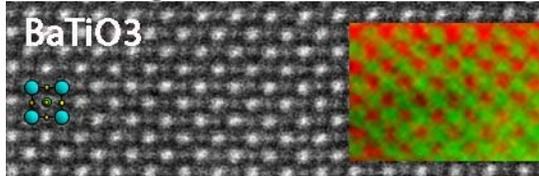


HD-2700C at BNL's CFN

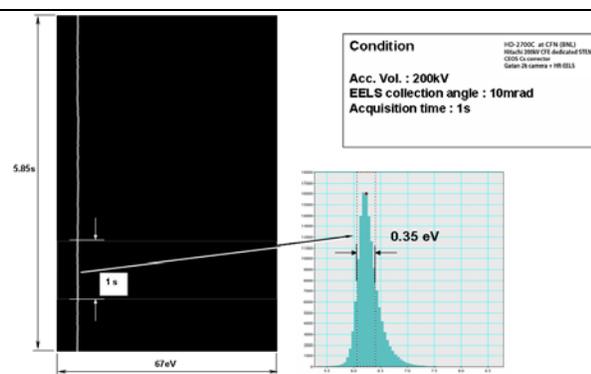
- *Remote operation
- *Custom sample holders
 - specimen heating + cooling (Gatan)
 - 360 degree rotation (Hitachi*)
- *Two stage projection optics
- *Ultra-fast installation time:
 - (only 2 weeks from uncrate to resolution)



HAADF image of Si <110> (40 sec)

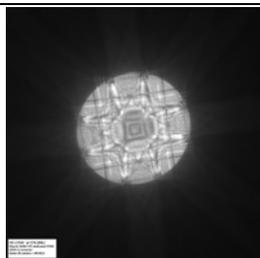


HAADF + EELS Spectrum Image (BaTiO3)



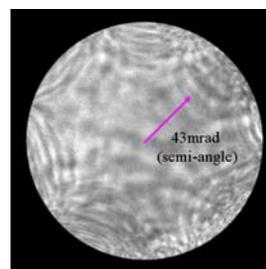
Erfinia-ER energy time trace of zero loss peak spectra for 5.85s to measure energy shift. Zero loss peak energy spectrum at 1s acquisition time of the time trace

Energy resolution and stability of CFE
(0.35 eV, 1 second acquisition time)
with beam-current (200pA / 2A)



STEM CBED and NBD (Si <100>)

> 40 mrad
(semiangle)



ABSTRACTS

DAY TWO

Atomic-Scale Studies of Complex Oxide Interfaces Using Aberration-Corrected Z-contrast Imaging and EELS

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Interfaces in complex oxide materials have been an enduring theme in materials physics, where the interplay of the reduced dimensionality, proximity effects, and surface relaxation and reconstruction creates interfacial states that are distinct from their bulk counterparts. It has been recognized that the perovskite oxides provide a unique opportunity to bring materials with diverse and mutually exclusive properties into intimate contact, and create interfaces with excellent structural and chemical compatibility and potentially novel properties.

In this presentation, aberration-corrected Z-contrast imaging and EELS in combination with in-situ heating/cooling experiments of interfaces in perovskite-type oxides will be shown. More specifically, we utilize the aberration-corrected JEOL2100FS at Brookhaven National Laboratory, the aberration-corrected VG HB-601UX at UIC and the conventional JEOL2010F TEM/STEM for our experiments in the temperature range between 10 K and 700 K. We will discuss our recent results, including the role of oxygen vacancies in the ultrathin SrTiO₃/GaAs hetero-interface, the effect of the spin-state transition of the Co³⁺ ions in LaCoO₃, and the effects of charge transfer in the Ca₃Co₄O₉.

The ultrathin SrTiO₃/GaAs interface consists of five monolayers of SrTiO₃(001) on GaAs (110), which are shown to be atomically flat without any obvious surface reconstruction and no significant diffusion between the film and the substrate.¹ While the SrTiO₃ film is highly oxygen deficient, the Fermi-level remains unpinned at the interface after a Ti-prelayer deposition.² Our ab-initio DFT calculation suggest that O-vacancies at the hetero-interface compensate the dangling bonds from the unreconstructed As-terminated GaAs (110) surface, and therefore play an important role in engineering the interfacial properties of this novel high-k dielectric.

Our temperature study of LaCoO₃ shows the effect of the Co³⁺-ion spin-state transition, which occurs at T_s~90K, on the fine-structure of the O K-edge. While the crystal structure of LaCoO₃ does not change during the in-situ cooling experiment to 10K, the O K-edge pre-peak intensity decreases for T>T_s. A detailed analysis of the O K-edge fine-structure and comparative DFT calculations will be presented.³

The misfit-layered structure Ca₃Co₄O₉, consists of triple rock salt-type layers Ca₂CoO₃ and single CdI₂-type CoO₂-layers stacked along the *c*-axis.⁴ We will present aberration-corrected Z-contrast imaging and atomic-column resolved EELS in conjunction with multiple scattering calculations to elucidate the effects of charge transfer between the different Co-layers and its effect on the thermoelectric properties of Ca₃Co₄O₉. Detailed analysis of this structure and the effects of in-situ heating and cooling on the Co-valence and spin-state will be discussed.

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Imaging and Exit Wave Reconstruction in an Aberration-Free Environment

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Electron-Optical aberration correctors are now firmly established as a key component in many commercial Transmission and Scanning Transmission Electron Microscopes installed around the world. Equally, algorithms that recover the complex specimen exit plane wavefunction from a series of images recorded using one of several possible experimental geometries have also advanced to the point where several commercial software implementations utilizing this approach are readily available.

This paper will discuss the combination of these two complementary approaches with particular reference to exit wave reconstruction from a series of tilted illumination images recorded for several azimuths. This latter geometry benefits substantially from initial electron optical aberration correction which enables larger tilt angles to be used during data acquisition. Direct correction of the spherical aberration also relaxes the otherwise stringent relationship that conditions the defocus and illumination tilt. I will discuss recent calculations that define optimal conditions for tilt azimuth based reconstructions with reference to the effects of higher order aberrations beyond those that can be corrected directly as well as the effects of finite chromatic aberration and sample thickness. It will be shown that overall these effects combine to allow optimal tilt angles of up to ca. 17mrad to be used (at 200kV) compared to ca. 4mrad in uncorrected instruments with a consequent improvement in the resolution of the reconstructed data.

In addition to the above theoretical considerations exit wave reconstruction under aberration corrected conditions also places stringent requirements on instrumental stability. We have recently installed our second generation double corrected 200kV instrument (JEOL JEM 2200MCO) in Oxford. This microscope incorporates both imaging and probe forming aberration correctors in addition to a number of design improvements leading to increased stability. Initial data from this machine will be presented demonstrating axial information transfer at <0.8nm in TEM.

Catalyst Nanoparticles Imaged Using Aberration Correction, Focal Series Restoration and Electron Tomography

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Industrial catalysts usually comprise crystalline particles of high atomic number that have sizes of between 1 and 20 nm and are supported or embedded in a lower atomic number matrix. In this talk, I will present a selection of recent results that illustrate how modern transmission electron microscopy techniques can be used to provide quantitative and useful information about 5-10 nm platinum catalyst nanoparticles supported on carbon. I will concentrate on the use of:

1. Spherical aberration correction and focal series restoration to improve image quality, visibility and interpretability;
2. High-angle annular dark-field electron tomography to characterize the three-dimensional morphologies, sizes and positions of the particles and the surrounding carbon support;
3. Low-angle annular dark-field imaging to assess the crystallinity of the particles and its influence on measurements of particle size distributions;

I will conclude by discussing the prospects offered by combining these and other techniques.

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Atomic-Scale Chemical Imaging by Aberration-Corrected Microscopy

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Using a fifth-order aberration-corrected scanning transmission electron microscope, which provides a factor of 100x increase in signal over an uncorrected instrument, we demonstrate two-dimensional elemental and valence-sensitive imaging at atomic resolution using electron energy loss spectroscopy, with acquisition times of well under a minute.

Applying this method to the study of a $\text{La}_{0.7}\text{Sr}_{0.3}\text{MnO}_3/\text{SrTiO}_3$ superlattice, we find an asymmetry between the chemical intermixing on the Mn/Ti and the La/Sr sublattices and corresponding changes in the formal valence. The changes in the Mn and Ti bonding as the local environment changes allow us to distinguish true chemical interdiffusion from imaging artifacts.

Cs Correction and Focal-Series Reconstruction of the Exit-Surface Wave

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The current revolution in nanoscale science and technology requires instrumentation for observation and metrology of nano-devices operating at the level of a few atoms, calling for microscopes with sufficient resolution for accurate atomic-scale imaging [1]. Sub-Ångström resolution in the high-resolution transmission electron microscope (HRTEM) has been achieved in the last few years by hardware aberration correction [2], software aberration correction [3-4], and electron holography [5].

Focal-series reconstruction (FSR) of the exit-surface electron wave in a non-hardware-corrected FEG-TEM has been used to produce Cs-corrected images with resolution out to the information limit of the microscope [6]. Realization of such resolution requires meticulous attention to experimental conditions, including careful balance of the tradeoff between high-frequency transfer and Cs-induced dispersion leading to large delocalizations that can place significant limits on the reconstructable field of view. In a hardware-corrected microscope, application of FSR is straightforward due to the low delocalization, allowing very accurate reconstruction of the exit-surface electron wave over large fields of view.

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Nanoscale Elemental Mapping in TEM and STEM

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For more than a decade elemental mapping by electron microscopy at resolutions approaching 1nm has been a core capability at the ORNL Shared Research Equipment (SHaRE) User Facility. Quantitative composition mapping has been emphasized and valence or bonding information has been less frequently studied. Two techniques have been used: energy-filtered transmission electron microscopy (EFTEM) and spectrum imaging in the scanning transmission electron microscopy (STEM) mode, often with simultaneous energy-dispersive X-ray spectroscopy (EDS) and electron energy-loss spectroscopy (EELS). Results of applications to materials such as nanostructured ferritic alloys, thin-film magnetic recording media based on Co-Cr and Co-Pt, Y-Ni-O nanocomposites, ceria abrasives, and nanostructured spinel, will be used to illustrate the advantages and limitations of the methods. The impact of recent and expected developments in instrumentation such as aberration correctors will be discussed.

Research supported by the Laboratory Directed Research and Development Program of ORNL, by the Office of Fusion Energy Sciences, by the Office of Nuclear Energy, Science and Technology through I-NERI 2001-007-F and at the ORNL SHaRE User Facility by the Division of Scientific User Facilities, Office of Basic Energy Sciences, U.S. Department of Energy, under Contract DE-AC05-00OR22725 with UT-Battelle, LLC. Collaborations with Drs. J.F. Al-Sharab, C.B. Carter, L. Chaffron, F. Cosandey, N.D. Evans, S.R. Gilliss, D.T. Hoelzer, S. McKernan, J.D. Risner, R. Sinclair, Z.L. Wang and J.E. Wittig are gratefully acknowledged.

Catalyst Characterization via Aberration-Corrected STEM in the HTML User Program

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The High Temperature Materials Laboratory (HTML) at Oak Ridge National Laboratory provides access to advanced instrumental techniques for materials characterization to university and industrial researchers. A capability to characterize materials structures at sub-Ångström resolution levels is provided by the JEOL 2200FS-AC STEM/TEM instrument, which is equipped with a hardware corrector (CEOS GmbH, Heidelberg, Ger.) on the incident probe, to allow high-angle annular dark-field images to be recorded. The instrument is housed in ORNL's new Advanced Microscopy Laboratory (along with 3 other aberration-corrected instruments), which has a controlled environment to allow routine operation of the instrument at its specified resolution. Coupled with the HTML's *ex-situ* catalytic reactor capabilities, the new microscope is particularly useful for imaging metal particles on catalysts, in studies such as the behaviour of Pt atoms and clusters on a variety of oxide supports when reduced, the core-shell structure of Au-Pd bimetallic nanoparticles, Ag on alumina catalysts for advanced lean NO_x applications, and ordering effects in bimetallic Pt-Co catalysts for fuel cell applications. Results from these studies will be discussed, and future directions for *in-situ* studies at the atomic level will also be reported.

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Aberration-Corrected STEM of MoVTenbO Complex Oxide Catalysts

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Acrylonitrile is an important intermediate in the production of a wide variety of important chemicals [1-3]. The current commercial methods for the production of acrylonitrile use propene over multi-component Bi-Mo-O_x catalysts [1]. Due to the increasing cost of propene, there is significant interest in replacing the more expensive propene with propane as a feedstock. The difficulty in switching to propane is the development of a multifunctional catalyst that is able to activate first a propane C-H bond and subsequently complete the ammoxidation to acrylonitrile without complete combustion to CO_x + H₂O, or other intermediate oxidation products. The dominant candidate for this next-generation process is based on the multiphase MoVTenbO complex oxide system [1-3].

The best MoVTenbO_x catalysts with respect to selectivity and activity is a two-phase mixture comprised of an orthorhombic network bronze phase (M1) and a hexagonal tungsten bronze (HTB)-type phase (M2) [2,3]. Transmission electron microscopy was initially used to identify unit cells and symmetry, and to obtain HREM images of the two phases. Starting with preliminary metal coordinates and initial guesses of the oxygen coordination, simultaneous Rietveld refinement of high resolution neutron and X-ray powder diffraction data led to models for both structures in which occupancies and valences were identified [3]. The Rietveld refinement of M1 involves a fit with roughly 200 adjustable parameters; the M2 structure is somewhat less complex, but still required simultaneous refinement of XRD and NPD data sets.

In this work we use Cs-corrected STEM imaging to further probe the structure of the M1 and M2 phases to provide direct evidence confirming the refined model structures. We show that the hexagonal channels in the M1 structure are filled with a high-Z cation in accordance with the refined model which incorporates Te into the hexagonal channels. In addition, some of the heptagonal channels are filled, again in accordance with the refined structure model.

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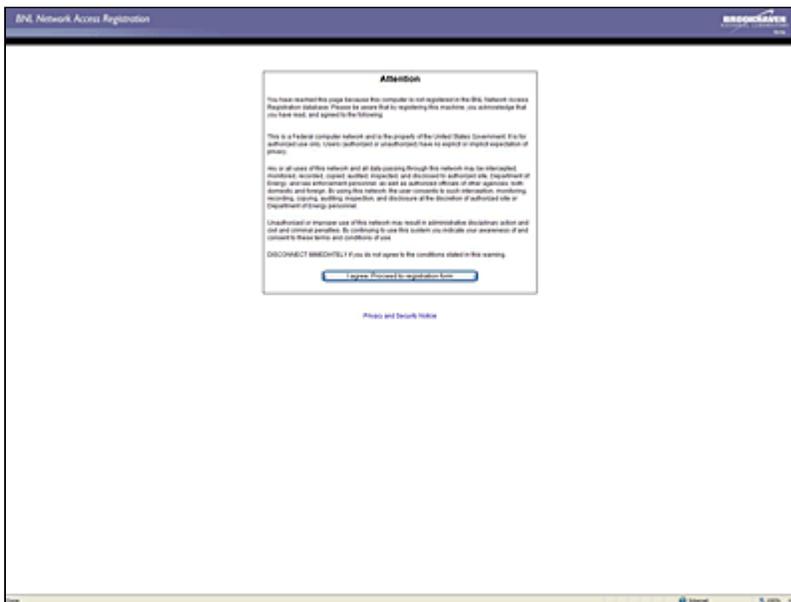
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Attendee Instructions - BNL policy requires that all computing devices connected to BNL's network be registered in Brookhaven's Network Access Registration system. If you will be using a computer (laptop, PDA, etc.) to access the internet or any of BNL's networked resources while on-site, and that computer has not been previously registered at BNL, your web browser traffic will be redirected to a web page (see below) requesting information about you and your computer. This form **MUST** be completed and submitted **within 30 minutes** of booting their computer, or your network access **will be** terminated. To begin this process, simply open your web browser once you have connected.

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Read notice and press the [I agree...] button to continue.



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Complete all required fields as directed in the accompanying sheet, then press the [Submit] button to finish. Please contact your Event Coordinator, or the on-site Helpdesk (laboratory extension 5522) if you experience problems.

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If you have reached this page, this computer is not registered for use on the BNL network. Please complete the registration form below to the best of your ability. An ACCESS KEY will be emailed with an administrator's email ID. If you will not be granted access to BNL network resources, upon submission of the form, you will be prompted to request Network Access with their work as requested.

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Owner/Primary User's Name* (last name, first name) <input type="text"/>	Administrator's Name (last name, first name) <input type="text"/>
Owner/Primary User's Phone* <input type="text"/>	Administrator's Phone <input type="text"/>
Owner/Primary User's E-Mail* <input type="text"/>	Administrator's E-Mail <input type="text"/>
Owner/Primary User's Life/Access Number* <input type="text"/>	Administrator's Life/Access Number <input type="text"/>
Owner/Host's Department* <input type="text"/>	
Building Number* (optional location of the computer) <input type="text"/>	Room Number/Name* (e.g. 102 or seminar room) <input type="text"/>
Host Number (e.g. 112-01-010) <input type="text"/>	System Type* <input type="text"/>
Operating System* <input type="text"/>	Secondary Operating System <input type="text"/>
Network Ownership <input type="radio"/> Personal <input type="radio"/> Other Non-BNL organization <input type="radio"/> Other Department at Energy entity <input type="radio"/> BNL (Default unless transfer) <input type="text"/>	Hardware (e.g. Dell Optiplex, HP Compaq, etc.) <input type="text"/>

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To aid you in the registration process, below is a list of the required fields and the information you will need to enter. Completion of fields **not** listed in these instruction is preferred, but not required.

Owner/Primary User Name:

Enter your name in LastName-comma-FirstName format (e.g. Doe, John).

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Enter: 3227

Owner/Primary User E-mail:

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Owner/Host Life/Guest Number:

Enter the following conference key: NC-58384

Department:

Select CENTER FOR FUNCTIONAL NANOMATERIALS from the drop down list provided.

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System Type:

Select the system type that best fits your device. If you're unsure, leave the default of WORKSTATION.

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Select your computer's operating system from the list provided. If you are unsure, you may select UNKNOWN. If your operating system is not listed, select OTHER.

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Workshop on Advanced Electron Microscopy in Materials Physics

Date : November 7-8th, 2007

Location: CFN, Brookhaven National Laboratory (NY)

This two day workshop will feature presentations by internationally prominent scientists working at the frontiers of electron microscopy, both on instrumentation development and applications in materials science.

The workshop is structured to stimulate scientific exchanges and explore new capabilities. Join your colleagues at BNL's world class facilities for an exciting two days of invited talks, informal discussions and technical exchanges. While the scientific themes of the workshop are focused on aberration corrected STEM and EELS, other advanced electron microscopy methods for application of materials physics are also incorporated. Invited speakers include P. Batson, R. Borkowski, N. Browning, M. Haider, A. Kirkland, B. Kabius, D. Muller, S. Pennycook, H. Rose, M. Ruehle and R. Trump.

There will be an opportunity check out the newly installed cold field emission, probe-corrected, dedicated-STEM equipped with an ultra-fast high resolution EELS, and tour the new Center for Functional Nanomaterials as well!

TO ATTEND, please REGISTER: <http://www.bnl.gov/cfn/seminars/>

- (1) Registration fee: \$100 – Registration deadline: Oct.15, 2007
(60 attendee limit, registration will be first-come first serve)
- (2) 30 days lead-time required for visitor access to Brookhaven National Lab!
(if you do not have a US passport, please contact: cfnuser@bnl.gov ASAP)
- (3) Conference rate at hotels near BNL, shuttle will be provided from the hotel.
(please contact NSDEvent@hitachi-hta.com for hotel info before Oct. 15th)

A complete schedule of speakers and topics can be viewed on the web at:

www.hitachi-hta.com/emd

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MEETING ANNOUNCEMENT
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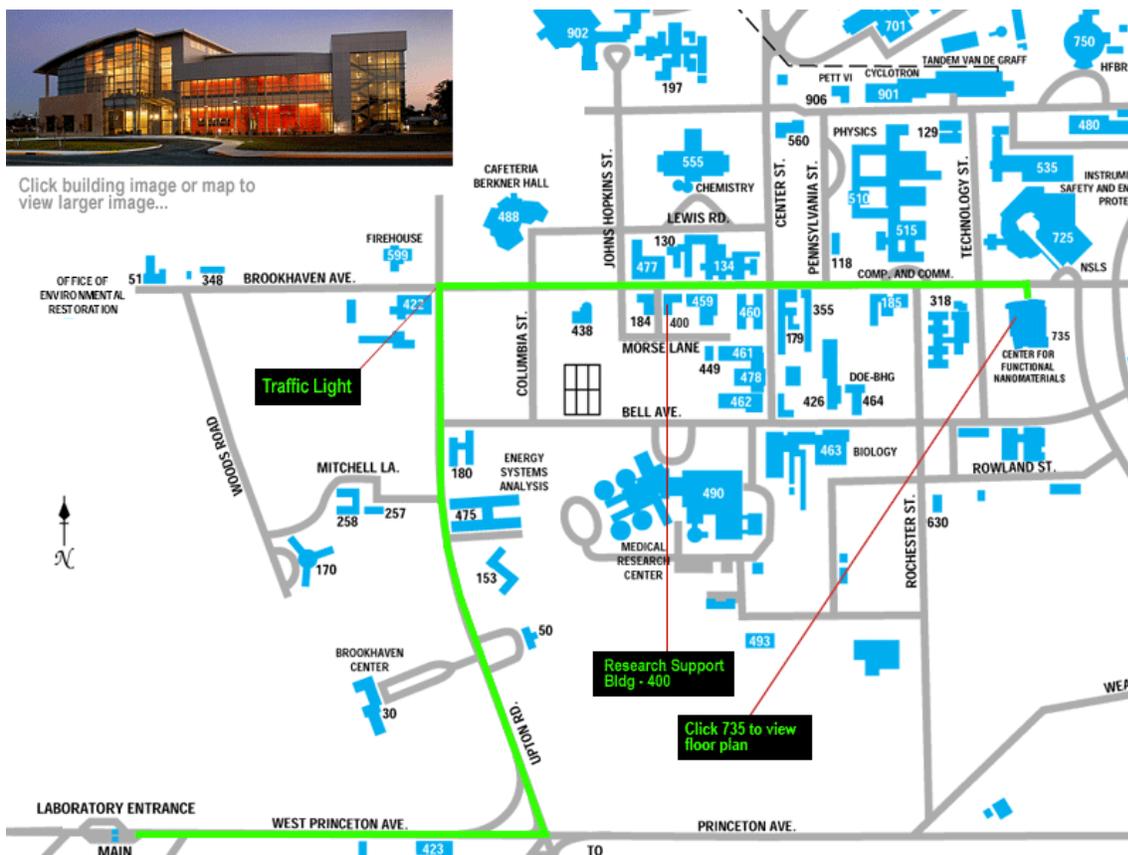


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Building 735

Directions to the Center for Functional Nanomaterials (CFN)

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The CFN will be located on Brookhaven Avenue across from the NSLS Building. Click the building image or map insert to view a larger scale or these imagers.

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